



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant : Brian H. Harrison et al.
Serial No.: 10/666,976
Filed: September 18, 2003
Title: RUBBER REDUCTION
Docket No.: 36115

LETTER

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

Enclosed is a certified copy of Canadian Patent Application No. 2,426,253; the priority of which has been claimed in the above-identified application.

Respectfully submitted,

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Date: 12-18-03

I hereby certify that this correspondence is being deposited with the United States Postal Service as first class mail in an envelope addressed to: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450 on the date indicated below.

John P. Murtaugh

Name of Attorney for Applicant(s)

12-18-03 John P. Murtaugh
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Specification and Drawings, as originally filed, with Application for Patent Serial No:
2,426,253, on April 22, 2003, by **BRIAN H. HARRISON** and **HURDON A. HOOPER**,
for "Rubber Reduction"

L. Lachance
Agent certificateur/Certifying Officer

September 19, 2003

Date

Canada

(CIPO 68)
04-09-02

OPIC  CIPO

Rubber Reduction

Field of the Invention

5 The invention generally relates to rubber reduction, and more particularly to tire reduction using a solvent at a subcritical temperature.

Background of the Invention

10 It is estimated that in many countries the number of used tires produced per year is approximately equal to the population of the country. As an example there are more than 250 million used tires produced annually in the United States. Methods of dealing with these used tires can generally be placed in two categories i.e. disposal and
15 reclamation. The former group includes land filling and stock piling which are increasingly unacceptable options for a multitude of reasons. Within the latter group are approaches that use the tires in close to their original state with possibly some physical processing. Examples within this group include vibration and debris dampening mats as may be used drilling operations or filler material for road construction. Most of the
20 applications in this category represent a limited volume of tires and do not exploit the economic value of the material within the tire.

 A second group of reclamation methods look to extracting value from the constituent materials within a tire. Major constituents include synthetic and natural rubber, carbon black and steel. Minor constituents include sulphur that is used for cross-
25 linking of the rubber chains during the vulcanization process. The most basic reclamation alternative is the use of tires as a fuel. In this case the tire may be in its original unprocessed state or it may have been reduced to simpler hydrocarbons such as oils. This latter alternative is favourable as constituents that are not desirable during burning would generally be removed. However, the use of tires as a fuel again reduces
30 the value of the reclaimed material to a point where there is little or no retention of value added during the fabrication of the tire.

 Therefore, there is a need for a method and system that provides for the reclamation of tires while maintaining value added to the constituent materials during the
35 original processing of the tire.

Summary of the Invention

5 The invention is directed to a method and system for the reduction of tires. An object of the invention is to provide a system and method for the reduction of tires to hydrocarbons where at least a portion of these hydrocarbons have a molecular weight that is larger than oils.

10 Other aspects and advantages of the invention, as well as the structure and operation of various embodiments of the invention, will become apparent to those ordinarily skilled in the art upon review of the following description of the invention in conjunction with the accompanying drawings.

Brief Description of the Drawings

15 The invention will be described with reference to the accompanying drawings, wherein:

20 Fig. 1a is a schematic graph illustrating % completion v. time for a pyrolysis reduction process;

 Fig. 1b is a schematic graph illustrating % completion v. time for a non-pyrolysis reduction process;

25 Fig. 2 is a flow chart of a process for reducing tires according to an embodiment of the invention;

 Fig. 3 is a flow chart of a process for reducing tires according to another embodiment of the invention; and

30 Fig. 4 is a flow chart of a process for reducing tires according to a further embodiment of the invention.

 Like numerals identify like features within the drawings.

35 Detailed Description of the Invention

The reduction of tires can generally be considered to include two steps or processes. The first of these is devulcanization where cross-linking sulphur bonds between rubber molecules are broken. At the end of the devulcanization process the mass of solid residue is approximately 100% of the original mass of tire. The solid residue contains rubber hydrocarbon and carbon black where rubber hydrocarbon includes any hydrocarbon originating from the initial rubber. The rubber hydrocarbon has an average molecular weight that is generally less than the initial rubber and greater than oil, where oil has an average molecular weight of approximately 500 or less. The depolymerization process reduces the average molecular weight of the rubber hydrocarbon until, at completion, the rubber hydrocarbon is substantially oil. The mass of solid residue is approximately 40% of the initial mass of tire at the end of the depolymerization process. At this point the solid residue is substantially only carbon black.

Figure 1a is schematic graph of the % completion v. time for a normal pyrolysis process. In a normal pyrolysis process a devulcanization process 102 and a depolymerization process 104 occur substantially in parallel. As such the two processes are complete at approximately the same time i.e. $t_v \approx t_p$. Figure 1b shows a non-pyrolysis process in which the devulcanization process 106 is separated from the depolymerization process 108. At time t_v the devulcanization process 106 is complete while the depolymerization process 108 is only a fraction of the way to completion.

The process of reducing tires according to an embodiment of the invention allows for at least some separation of the devulcanization and a depolymerization processes. A flow chart of a reduction process according to an embodiment of the instant invention is presented in Figure 2. Tire feedstock is provided to a reactor suitable for temperatures and pressures appropriate for subcritical treatment with water at step 202. Water is provided to the reactor at step 204. The amount of water provided is sufficient to maintain the saturated water vapour pressure at the reaction temperature and to allow the tire feedstock to be immersed in the liquid water at the reaction temperature.

The reactor is heated to the reaction temperature at step 206. To maintain a subcritical reaction the reaction temperature is less than the critical temperature of water. The reaction pressure is provided at step 208. This pressure is greater than or equal to the saturated water vapour pressure at the reaction temperature. In the current embodiment the reaction temperature is between about 280°C and about 350°C and the reaction

pressure is between about 950 psig and about 2400 psig. The reaction is continued at step 210 for time t. In the current embodiment time t is less than about 1 hr. A reaction product is produced at step 212. The reaction product produced according to the process of the current embodiment includes devulcanized rubber hydrocarbon, carbon black and may include some oils resulting from at least partial depolymerization of the rubber hydrocarbon.

A flow chart of a reduction process according to another embodiment of the invention is presented in Figure 3. The process includes the steps presented in Figure 2. The process according to this embodiment of this invention further includes the step of treating the reaction product with a solvent at step 302. In this embodiment the solvent is one appropriate for the dissolution of rubber hydrocarbon, including toluene and cyclohexane. The use of other solvents and mixtures of solvents as appropriate for the dissolution of the reaction product, which may include hydrocarbons of varying molecular weight will be apparent to one skilled in the art. Carbon black is then separated from the reaction product at step 304 using filtration methods. It will be apparent to one skilled in the art that any appropriate techniques may be implemented for the separation of the carbon black from the dissolved reaction product.

A flow chart of a reduction process according to a further embodiment of the invention is presented in Figure 4. The process includes the steps presented in Figure 3. The process according to this embodiment of this invention further includes the step of separating the solvent from the rubber hydrocarbon. Techniques appropriate for separating the solvent from the rubber hydrocarbon will be apparent to one skilled in the art. In this embodiment evaporation is used to separate the solvent from the rubber hydrocarbon.

Process parameters for subcritical fluid techniques include pressure, temperature, fluid or solvent and time. Without being bound by theory it appears that operation of the process at temperatures below the critical temperature of the solvent allows for the separation of the devulcanization and the depolymerization processes. The rate of depolymerization process is lowered to an extent where it is effectively separated from the step of devulcanization. This allows improved control regarding the extent to which depolymerization is allowed to proceed. In an embodiment of the invention the reaction time is varied to provide substantially complete devulcanization and a desired amount of depolymerization.

5 It will be apparent to one skilled in the art that tires can include any vulcanized rubber tire as may be used for transportation purposes. It will also be apparent to one of skill in the art that other vulcanized rubber, synthetic and natural, products may be used as a feedstock.

10 It will be further apparent to one of skill in the art that the processing conditions presented above are with respect to one embodiment of the invention. The invention encompasses those processing parameters i.e. temperature, pressure, time and solvent that allow for the separation of the devulcanization and depolymerization steps within the reduction process.

15 While the invention has been described according to what is presently considered to be the most practical and preferred embodiments, it must be understood that the invention is not limited to the disclosed embodiments. Those ordinarily skilled in the art will understand that various modifications and equivalent structures and functions may be made without departing from the spirit and scope of the invention as defined in the claims. Therefore, the invention as defined in the claims must be accorded the broadest possible interpretation so as to encompass all such modifications and equivalent structures and functions.

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What is claimed is:

1. A process for reducing rubber, the process comprising the steps:
5 providing the rubber to a reactor;
 providing a solvent to the reactor;
 elevating a temperature within the reactor to a temperature wherein the
temperature is below a critical temperature of the solvent;
 elevating a pressure within the reactor to a pressure of at least the saturated
vapour pressure of the solvent at the temperature;
10 reducing the rubber for a specified time; and
 producing a reaction product.
2. A process as claimed in claim 1 wherein the step of reducing the rubber comprises the
15 steps of:
 devulcanizing the rubber to produce rubber hydrocarbon;
 depolymerising the rubber hydrocarbon to produce hydrocarbons with a lower
molecular weight than a molecular weight of the rubber hydrocarbon.
3. A process for reducing rubber, the process comprising the steps of:
20 providing rubber to a reactor;
 providing a solvent to the reactor;
 elevating a temperature within the reactor to a temperature wherein the
temperature is below a critical temperature of the solvent;
 elevating a pressure within the reactor to a pressure equal to the saturated vapour
25 pressure of the solvent at the temperature;
 reducing the rubber for a specified time; and
 producing a reaction product.
4. A process as claimed in claim 3 wherein the step of reducing the rubber comprises
30 the steps of:
 devulcanizing the rubber to produce rubber hydrocarbon;
 depolymerising the rubber hydrocarbon to produce hydrocarbons with a lower
molecular weight than a molecular weight of the rubber hydrocarbon.
- 35 5. A process as claimed in any of claims 1 to 4 wherein the rubber is tire.

6. A process as claimed in any of claims 1 to 5 wherein the solvent is water.

7. A process as claimed in claim 5 wherein the temperature is between about 280°C and about 350°C.

5

8. A process as claimed in claim 6 wherein the pressure is between about 950 psig and about 2400 psig.

9. A process according to any of claims 1 to 8 wherein the specified time is less than about one hour.

10

10. A process according to any of claims 1 to 9 wherein the rubber is within the liquid solvent during the reaction.

11. A process according to any of claims 1 to 10 wherein the reaction product comprises rubber hydrocarbon and carbon black.

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12. A process according to any of claims 1 to 11 wherein the reaction product comprises substantially no sulfur.

20

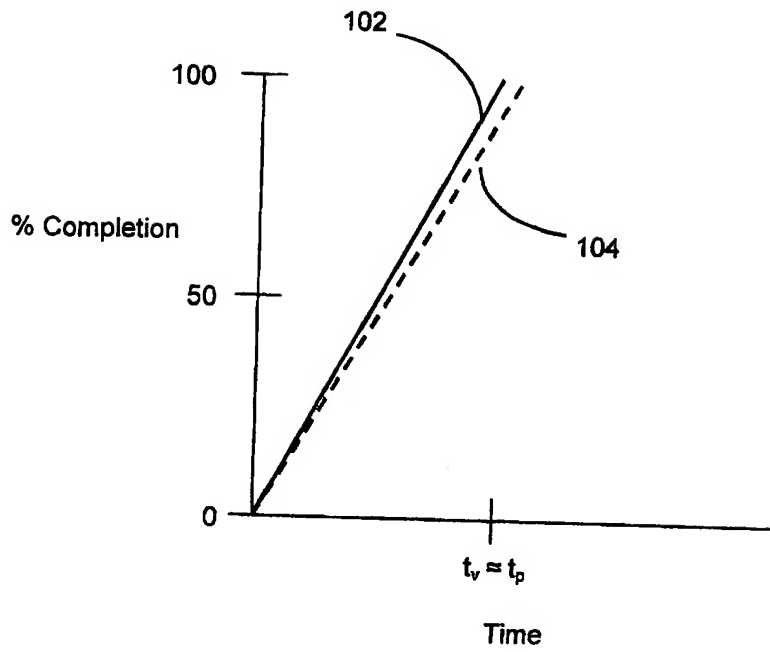


Figure 1a

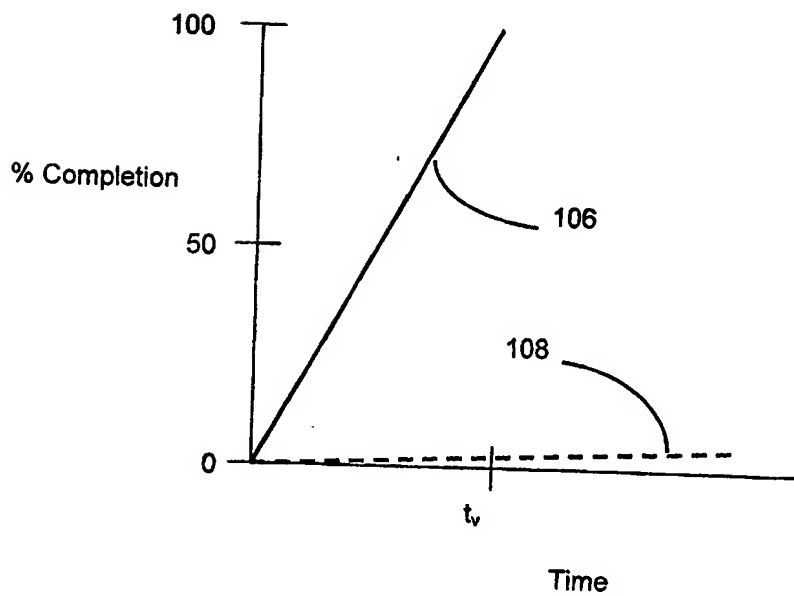


Figure 1b

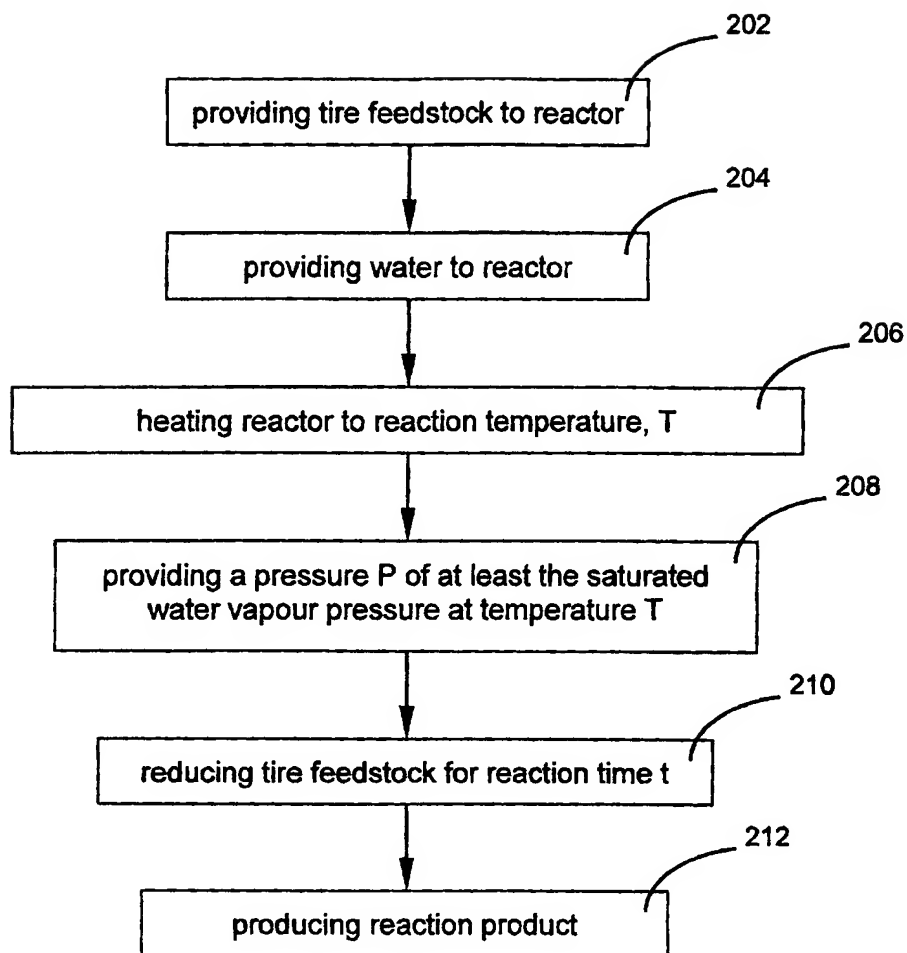
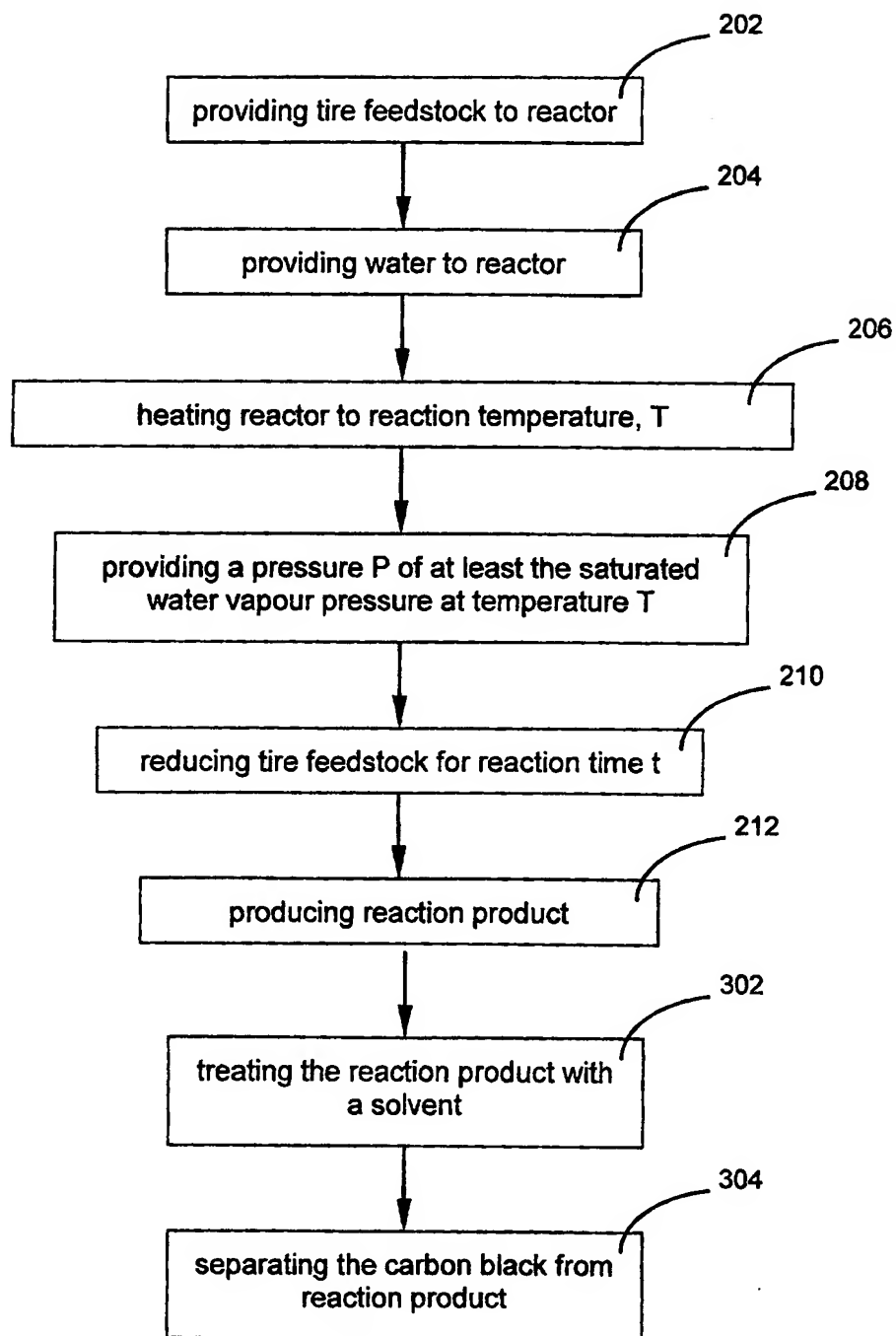
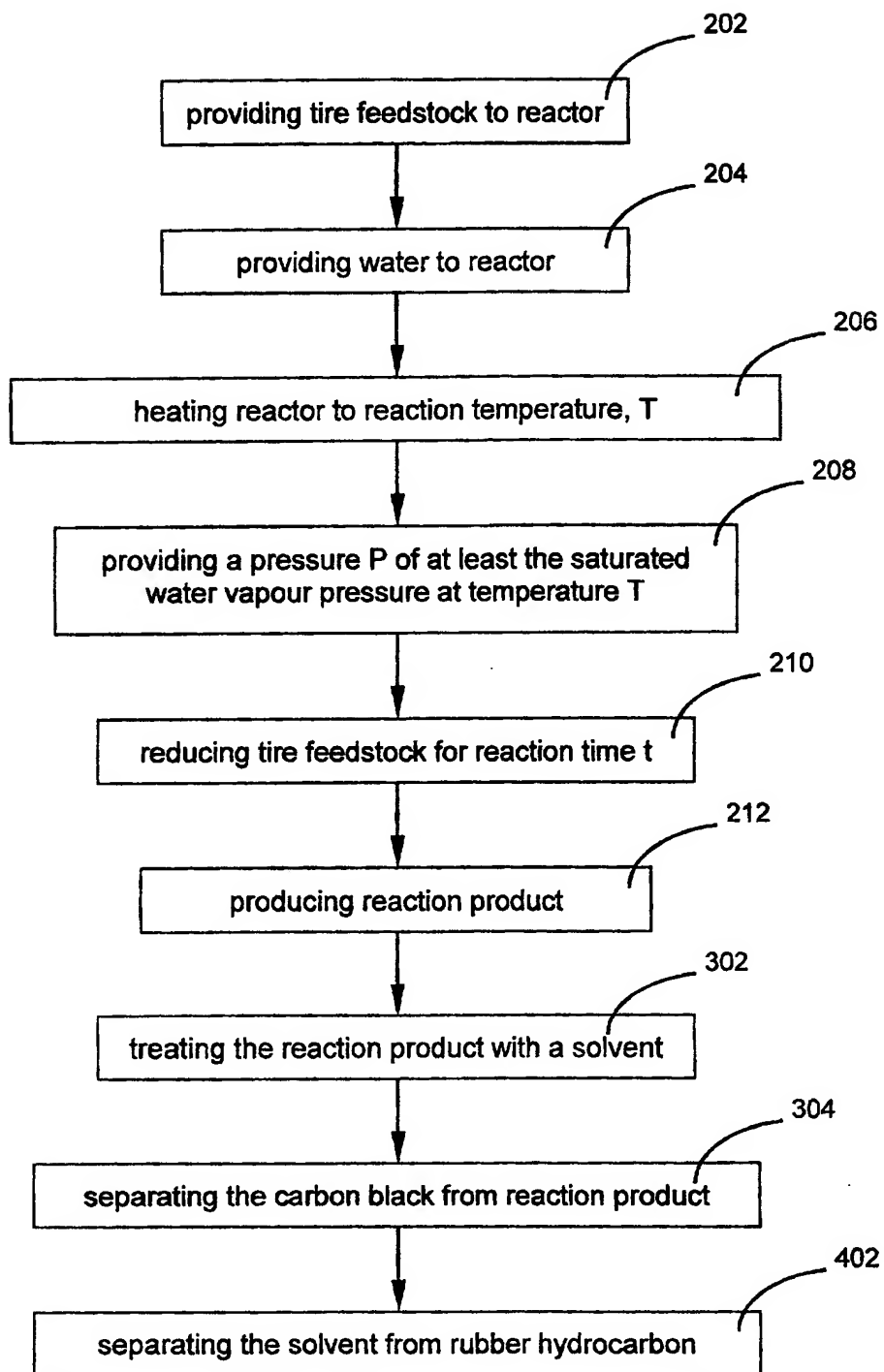


Figure 2

Gowling Lafleur Henderson LLP

**Figure 3**

**Figure 4**

Abstract

5 A process for reducing rubber is provided. The process includes the steps of providing the rubber to a reactor, providing a solvent to the reactor, elevating a temperature within the reactor to a temperature wherein the temperature is below a critical temperature of the solvent, elevating a pressure within the reactor to a pressure of at least the saturated vapour pressure of the solvent at the temperature, reducing the rubber for a specified time and producing a reaction product.